

### **REMARKS**

The above amendments to the above-captioned application along with the following remarks are being submitted as a full and complete response to the Official Action dated June 18, 2004 and the phone interview with the Examiner on September 15, 2004. Applicants thank Examiner for taking the time to conduct the interview.

In view of the above amendments and the following remarks, the Examiner is respectfully requested to give due reconsideration to this application, to indicate the allowability of the claims, and to pass this case to issue.

### **Status of the Claims**

Claims 1, 2, 5-7, 9-11, 13 and 15-18 are under consideration in this application. Claims 4, 8, 12 and 14 are being cancelled without prejudice or disclaimer. Claims 1 and 7 are being amended, as set forth in the above marked-up presentation of the claim amendments, in order to more particularly define and distinctly claim applicants' invention. New claims 15-18 are being added to recited other embodiments of the invention.

### **Additional Amendments**

The claims are being amended to correct formal errors and/or to better disclose or describe the features of the present invention as claimed. All the amendments to the claims are supported by the specification. Applicants hereby submit that no new matter is being introduced into the application through the submission of this response.

### **Prior Art Rejections**

Claims 1-2, 4-6 and 9 - 12 were rejected under 35 U.S.C. § 103(a) on the grounds of being unpatentable over an article by Honda et al (IEEE Transactions on Magnetics, Vol. 36, No. 5, Sept. 2000; pp. 2399-2401; hereinafter "Honda") in view of US Pat. No. 6,120,890 to Chen et al. (hereinafter "Chen"). The Examiner also rejected claims 7 - 8 and 13 -14 under 35 U.S.C. § 103(a) on the grounds of being unpatentable over Honda et al. (IEEE) in view of Chen et al. '890 in further view of US Pat. No. 6,001,447 to Tanahashi et al (hereinafter

“Tanahashi”). These rejections have been carefully considered, but are most respectfully traversed.

The perpendicular magnetic recording medium according to the invention (e.g., Fig. 1), as now recited in claim 1, comprises: a substrate 11; a soft magnetic underlayer 13 formed on said substrate 11 and containing ferromagnetic  $\alpha$ -Fe nanocrystals (p. 3, 3<sup>rd</sup> and 4<sup>th</sup> lines from the bottom; p. 6, last four lines); a nonmagnetic intermediate layer 14 formed on said soft magnetic underlayer 13; and a perpendicular recording layer 15 formed on said intermediate layer 14. The soft magnetic underlayer 13 contains ferromagnetic  $\alpha$ -Fe nanocrystals, Ta and C, and a concentration of said Ta ranges from 8 at % to 15 at %. In particular, a nonmagnetic amorphous or nanocrystalline pre-coating layer 12 is provided between said substrate 11 and said soft magnetic underlayer 13 and comprises at least one selected from the group consisting of NiZr alloy, NiTa alloy, NiNb alloy, NiTaZr alloy, NiNbZr alloy, CoCrZr alloy, NiCrZr alloy (p. 4, lines 8-15).

The pre-coating layer 12 is maintained nonmagnetic amorphous or nanocrystalline between said substrate 11 and said soft magnetic underlayer 13 even if the soft magnetic underlayer 13 is provided with ferromagnetic  $\alpha$ -Fe nanocrystals by annealing at 450°C or more (p. 6, last paragraph; claims 15, 17).

The invention is also directed to a magnetic storage apparatus recited in claim 7 containing the perpendicular magnetic recording medium recited in claim 1.

Applicants respectfully contend that neither Honda nor any other cited prior art reference teaches or suggests such a laminated structure of a perpendicular magnetic recording medium having “a nonmagnetic amorphous or nanocrystalline pre-coating layer 12 comprising at least one selected from the group consisting of NiZr alloy, NiTa alloy, NiNb alloy, NiTaZr alloy, NiNbZr alloy, CoCrZr alloy, NiCrZr alloy and being maintained between said substrate 11 and said soft magnetic underlayer 13” according to the invention.

As admitted by the Examiner, Honda fails to teach an amorphous or nanocrystalline pre-coating layer formed between the substrate and the soft magnetic underlayer. The NiP sealing layer 21 of Chen was relied upon by the Examiner to teach an amorphous or nanocrystalline pre-coating layer formed between the substrate and the soft magnetic underlayer. Chen (Fig. 2; col. 6, lines 41-48) discloses a magnetic recording medium having a laminated structure comprising: a substrate 20; a NiP sealing layer 21; an underlayer 22; and

a magnetic layer 23 (in-plane or perpendicular). Chen's sealing layer 21 is composed of NiP or with "*one more elements, such as B, W and Nb* (col. 5, lines 40-45)", rather at least one selected from the group consisting of NiZr alloy, NiTa alloy, NiNb alloy, NiTaZr alloy, NiNbZr alloy, CoCrZr alloy, NiCrZr alloy.

Applicants contend that the presence of the pre-coating layer 12 made of these specific materials allows the soft magnetic underlayer 13 being a layer in which fine and uniform nanocrystals are precipitated, thereby reducing a medium noise caused by the soft magnetic underlayer 13 (p. 9, Table 3 to p. 10). In the present invention, the unexpected result of the ferromagnetic  $\alpha$ -Fe nanocrystals being fine and uniform, more specifically, uniform precipitated with the grain diameter of about 10 nm (p. 10, lines 7-8) after annealing was a result of the presence of the pre-coating layer 12 made of NiZr alloy, NiTa alloy, NiNb alloy, NiTaZr alloy, NiNbZr alloy, CoCrZr alloy under the soft magnetic underlayer 13. The presence of the unexpected properties is evidence of nonobviousness. MPEP§716.02(a).

Even if the NiP sealing layer 21 of Chen were provided in Honda as suggested by the Examiner, Chen's sealing layer 21 composed of NiP or with B, W and Nb simply does not result in such a soft magnetic underlayer containing therein ferromagnetic  $\alpha$ -Fe nanocrystals have fine and uniform particle diameters of approximately 10 nm as in the invention.

Further more, Honda teaches non-uniform polycrystalline grain sizes ranging from 5 to 20 nm in (III, Results and Discussion, line 6) which will be effected by thermal fluctuation thus produce negligible noise (due to some bigger particles and the dispersion of polycrystalline grain size). Concerning the relationship between the grain diameters of the ferromagnetic  $\alpha$ -Fe nanocrystals included in the soft magnetic underlayer and the characteristics of the soft magnetic underlayer, when the grain diameters are small and uniform as in the present invention, the Hc (coercivity) at 298 K decreases to 1 Oe or lower (p. 10, lines 9-10). When there is dispersion in grain diameters including large particles of 20 nm, as in Honda, the Hc (coercivity) at 298 K increases to, such as 5.4 Oe as shown in Table 1 B in Honda. It is well established that a rejection based on cited references having contradictory principles or principles that teach away from the invention is improper.

Moreover, the nonmagnetic amorphous or nanocrystalline pre-coating layer 12 comprising at least one selected from the group consisting of NiZr alloy, NiTa alloy, NiNb alloy, NiTaZr alloy, NiNbZr alloy, CoCrZr alloy, NiCrZr alloy of the invention is maintained between said substrate 11 and said soft magnetic underlayer 13 even if the soft magnetic

underlayer 13 is provided with ferromagnetic  $\alpha$ -Fe nanocrystals by annealing at 450°C or more.

Such an unique structure or the second unexpected result, was enabled by the pre-coating layer 12 made of *“a material which shows good adhesion with the substrate, has a flat surface and reacts a little with the soft magnetic underlayer by annealing should be used as the pre-coating layer. To be concrete, amorphous or nanocrystal materials such as NiZr alloy, NiTa alloy, NiNb alloy, NiTaZr alloy, NiNbZr alloy, CoCrZr alloy and NiCrZr alloy can be used. Herein, the amorphous and nanocrystal materials indicate one in which no clear diffraction peaks can be observed by  $\theta$ -2 $\theta$  scan mode of the powder X-ray diffraction and  $2\theta$  scan mode of the thin film X-ray diffraction (p. 4, lines 8-15)”*. Accordingly, the pre-coating layer 12 shows only a little reaction with the soft-magnetic underlayer 13 upon annealing at a thermal treatment (p. 4, lines 4-9). The non-uniformity of the soft magnetic properties due to the substrate material or the temperature distribution during annealing in the prior art is thus suppressed by (i) forming the amorphous or nanocrystalline pre-coating layer 12 on the substrate 11, (ii) crystallizing and forming the soft magnetic layer 13 on the nanocrystalline pre-coating layer 12 (*“the Fe-6at %Ta-12at %C film 13 is crystallized during a film formation (before annealing), while the as-deposited films 12 having the other compositions are substantially amorphous as shown in FIG. 3 (p. 8, lines 13-15)”*), and then (iii) providing the soft magnetic underlayer 13 with ferromagnetic  $\alpha$ -Fe nanocrystals therein by annealing at 450°C or more (p. 3, 3<sup>rd</sup> and 4<sup>th</sup> lines from the bottom; p. 6, last four lines). As such, the invention provides a perpendicular magnetic recording medium which sufficiently reduces medium noises attributable to the soft magnetic underlayer 13 (p. 4, lines 4-8).

Although the Examiner asserted that annealing of the FeTaC layer at a given temperature will not necessarily heat treat the NiP layer at the same temperature (p. 5, lines 6-7 of the outstanding office action), it is intuitive for one skilled in the art to anneal the FeTaC layer and heat-treat the NiP layer at the same temperature in view of Honda and Chen. As such, Ni in Chen's NiP sealing layer 21 is crystallized and magnetized in annealing the FeTaC layer, rather than maintaining amorphous or nanocrystalline when the soft magnetic underlayer 13 being formed and annealed as in the invention. One skilled in the art simply would not anneal the FeTaC layer and heat-treat the NiP layer at different temperatures as the invention based on the above prior teachings except by using Applicants' invention as a

blueprint. Applicants will point out that a rejection based on hindsight knowledge of the invention at issue is improper.

Applicants submit two pieces of evidence as follows to show that the combination of Chen's NiP sealing layer 21 with Honda's substrate and Fe-Ta-C soft magnetic underlayer would crystallize and magnetize Ni in Chen's NiP sealing layer 21 such that Chen's NiP sealing layer could not maintain its amorphous or nanocrystalline state. The intended purpose of inserting Chen's NiP sealing layer 21 would be totally destroyed in the combination. Applicants contend that one skilled in the art will not be motivated to combine the teachings in Honda and Chen in the manner suggested by the Examiner since the resulting physical change of Chen's NiP sealing layer 21 will contradict its intended purpose.

As shown in the article titled "Interfacial Effects and Novel Properties of Nanomaterials," submitted by fax on June 10, 2004 and resubmitted herewith (see underlined portions), Chen's amorphous NiP forming the sealing layer crystallizes upon heating at temperatures exceeding 360°C such that it can not maintain its amorphous state when heat-treated at 450°C along with Honda's FeTaC layer (p. 2399, left col. last paragraph, lines 6-7).

Material 1/ Chart 1 is submitted herewith to show the relationship between the crystalline state of the FeTaC layer and annealing temperature. It also shows the relationship between the crystalline state of the FeTaC layer, Bs and Hc. (1) Amorphous region: At temperatures of about 360°C, at which NiP is crystallized, although some nanocrystals start to be formed, most of the film is amorphous and so Bs is low (approximately 0.7 T) while Hc is large (approximately 1.5 Oe). Namely, good soft magnetic properties cannot be obtained. (2) Amorphous+nano-crystalline region: At 400°C, amorphous state and nanocrystals coexist in a most non-uniform state, resulting in a large Hc. (3) Nano-crystalline region: At 450°C or higher, uniform nanocrystals can be obtained, so that good soft magnetic properties ( $B_s > 1.6\text{ T}$ ,  $H_c < 0.2\text{ Oe}$ ) can be obtained. Thus, the soft magnetic underlayer containing ferromagnetic -Fe nanocrystals is realized only by annealing at 450°C or higher. The nano crystalline state, which is the desired crystalline state, cannot be obtained if the soft magnetic underlayer is annealed at temperatures lower than 360°C which NiP can maintain its amorphous state.

On the other hand, in the present invention, the amorphous or nanocrystalline pre-coating layer 12 is capable of maintaining its amorphous state and nonmagnetic even after a high-temperature annealing treatment for obtaining a soft magnetic underlayer 13 containing

ferromagnetic  $\alpha$ -Fe nanocrystals as discussed previously. Thus, the soft magnetic underlayer 13, which is formed on the amorphous or nanocrystalline pre-coating layer 12, is prevented from magnetic non-uniformity thereby significantly reducing media noise according to the invention (p. 3, lines 13-15).

Although the two unexpected properties were unknown and non-inherent functions in view of Honda and/or Chen, since they do not inherently achieve the same results. In other words, these advantages would not flow naturally from following the teachings of Honda and Chen, since they fail to suggest a nonmagnetic amorphous or nanocrystalline pre-coating layer 12 comprising at least one selected from the group consisting of NiZr alloy, NiTa alloy, NiNb alloy, NiTaZr alloy, NiNbZr alloy, CoCrZr alloy, NiCrZr alloy and being maintained between said substrate 11 and a soft magnetic underlayer 13 containing fine and uniform ferromagnetic  $\alpha$ -Fe nanocrystals.

Applicants further contend that the mere fact that one of skill in the art could rearrange Honda and Chen to meet the terms of the claims is not by itself sufficient to support a finding of obviousness. The prior art must provide a motivation or reason for one skilled in the art to provide the unexpected properties, such as providing a soft magnetic underlayer with fine and uniform ferromagnetic  $\alpha$ -Fe nanocrystals (the grain diameter of about 10 nm) even after annealing at 450°C or more thereby reducing a medium noise caused by the soft magnetic underlayer, without the benefit of appellant's specification, to make the necessary changes in the reference device.

Applicants contend that neither Honda nor Chen teaches or discloses each and every feature of the present invention as disclosed in at least independent claims 1 and 7. As such, the present invention as now claimed is distinguishable and thereby allowable over the rejections raised in the Office Action. The withdrawal of the outstanding prior art rejections is in order, and is respectfully solicited.

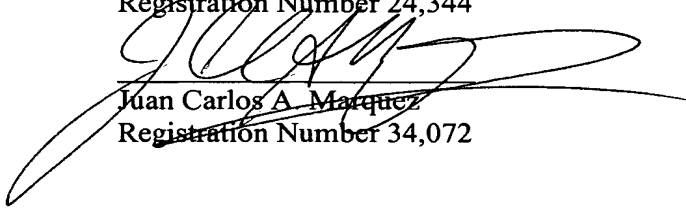
In view of all the above, clear and distinct differences as discussed exist between the present invention as now claimed and the prior art reference upon which the rejections in the Office Action rely, Applicants respectfully contend that the prior art references cannot anticipate the present invention or render the present invention obvious. Rather, the present invention as a whole is distinguishable, and thereby allowable over the prior art.

Favorable reconsideration of this application is respectfully solicited. Should there be any outstanding issues requiring discussion that would further the prosecution and allowance

of the above-captioned application, the Examiner is invited to contact the Applicants' undersigned representative at the address and phone number indicated below.

Respectfully submitted,

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## Interfacial Effects and Novel Properties of Nanomaterials

or here: Direct Link To Solid State Phenomena, Volume: 94

First part of the paper. Find the complete, well formatted version here: <http://3-908450-80-2.scientific.net/>

### Studies of Amorphous Ni-P Thin Films Produced by the Chemical Reduction Method, 3-908450-80-2

Studies of Amorphous Ni-P Thin Films Produced by the Chemical Reduction Method K. Konopka, M. Trzaska, D. Oleszak, W. Zieliński, K.J. Kurzydłowski Warsaw University of Technology, Faculty of Materials Science and Engineering, 02-507 Warsaw, Poland Keywords: Ni-P alloy, amorphous material, crystallization, microstructure Abstract. The paper is focused on the crystallization of Ni-P amorphous thin films produced by a chemical reduction method. The results of differential scanning calorimetric (DSC) experiments, X-ray diffraction analysis (XRD), scanning (SEM) and transmission electron microscopy (TEM) studies of the microstructures are presented. The surface morphology of Ni-P amorphous films can be described in terms of arrays of spherical particles and holes, related to H<sub>2</sub>, distributed mostly at their borders. The DSC studies of the films showed two exothermic effects at 370 and 440°C. This implies that crystallization proceeds in two stages. First, the metastable crystalline phase Ni<sub>12</sub>P<sub>5</sub> is formed and then transforms into the equilibrium phases Ni<sub>3</sub>P and Ni. After annealing at a temperature below the peak of crystallization, the amorphous structure is visible. However, near the edge of perforations of TEM thin foils, the presence of the nanocrystals were revealed and the holes disappeared. Further research of both phenomena, nanocrystallization and healing of holes, are in progress. Introduction The high strength and hardness of nanocrystalline materials [1,2] make them attractive for various applications and have provided an impetus for the development of novel methods for their production. One of them is the nanocrystallization of amorphous alloys. Amorphous Ni-P alloy has been extensively investigated in the last few years because of its high corrosion resistance and potential applications in catalysis [3,4,5]. Studies of the Ni-P system show various sequences of crystallization. Crystallization of the Ni<sub>3</sub>P phase was reported in [6]. Another metastable phase that transformed into the equilibrium phase Ni<sub>3</sub>P was described in [7]. The microstructures of Ni-P alloy after crystallization vary from an amorphous structure, to spherulitic crystalline regions surrounded by the amorphous matrix, and then to a mixture of crystalline Ni and tetragonal Ni<sub>3</sub>P. The crystalline regions in the early stages of crystallization of amorphous Ni-P have a nanometer size [8]. However, there is no commonly accepted model for the crystallization and further development of the microstructures after this process. It should be noted that the amorphous alloys are thermodynamically unstable. Their crystallization occurs spontaneously even below their crystallization temperature [3] and, therefore, it seems essential to carefully examine the changes of the microstructures in Ni-P just before and after crystallization. The results of such investigations can help to understand the mechanisms taking place during the heat treatment of amorphous alloys and the control of the nanocrystallization process. The paper focuses on the crystallization process of the Ni-P thin films produced by a chemical reduction method. The results of DSC experiments, X-ray diffraction analysis, SEM and TEM studies of the microstructures are presented. Experimental procedures The film of amorphous Ni-P alloy (12.5 at % of P) was deposited on glass by a catalytic chemical reduction. The process has been carried out in a multi-component electrolyte with NiCl<sub>2</sub> salt as the basic supply of the nickel ions. NaH<sub>2</sub>PO<sub>4</sub> was used as the reducer and C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>Na<sub>3</sub> fulfilled the role of a stabilization compound to maintain the pH of the solution between 4 and 5. The temperature of the bath was kept within the range 87 to 92°C. X-ray diffraction and DSC techniques were used for studying the crystallization processes of amorphous Ni-P films. A Philips PW 1830 diffractometer with CuK radiation was used for XRD investigations and DSC measurements were carried out in Perkin Elmer DSC 7 unit. The samples were continuously heated within the temperature range 50 to 600 °C at a heating rate of 20 °C min<sup>-1</sup>. The surface morphology and microstructures of the Ni-P samples were observed by LEO1530 scanning electron microscope and Philips 300 transmission electron microscope. Results Figure 1 shows a typical DSC curve



registered during continuous heating of the Ni-P films studied. Two exothermic effects are visible with peaks at temperatures of 370 and 440 oC. The temperature of isothermal annealing is indicated by the arrow in Figure 1. 270 C exo Heat Flow [a.u.] o o Fig.1. DSC curve of the Ni-P films Tp2= 440 C 20 C/min. 100 200 300 o Tp1= 370 C 400 o o 500 600 Temperature [ C] A set of XRD patterns of samples after various heat treatments is given in Figure 2. The pattern for the as received Ni-P samples is also included. The diffraction pattern of the as received starting sample reveals a diffused halo, which is typical for amorphous materials (Fig. 2a). Heating of the sample to 360 oC results in the crystallization of the amorphous phase, and first diffraction lines appear in the XRD pattern. A unique identification of this phase is not possible. The lines marked by arrows have been identified as indicating Ni12P5 phase (Fig. 2b). However, in view of the concentration of P in the alloy, the appearance of such a phase is improbable unless there is a segregation of P in the film, but the XRD technique does not reveal it. After heating of the sample to the peak temperature of 370 oC the diffraction lines of Ni3P, in addition to those of Ni12P5, are visible (Fig. 2c). Finally, the XRD pattern for the sample heated to 600 oC, i.e., fully crystallized, consists of the diffraction lines of Ni and Ni3P phases only (Fig. 2d). The surface morphology of the amorphous Ni-P films can be described in terms of arrays of spherical particles (Fig.3). These particles in the amorphous alloy are agglomerated into large "lumps" (Fig.4). TEM image of amorphous Ni-P film: black dots- the holes made by H2 black shadows- contours of particles Fig. 5. TEM image of the Ni-P film after annealing at 270 oC One more interesting feature of the microstructure obtained as a result of annealing is the absence of the holes which were visible in the amorphous films prior to heating. The disappearance of these holes may indicate the escape of hydrogen through the surface during the process of heating and, as a consequence of diffusion, "healing" of the material. Summary and Conclusions The DSC studies of Ni-P amorphous films showed two exothermic effects at 370 and 440 oC, which imply that crystallization proceeds in two stages. The crystallization of the amorphous material starts at 360 oC via formation of Ni12P5 phase. This non-equilibrium phase disappears at higher temperatures, where equilibrium Ni and Ni3P are formed. Two kinds of crystalline diffraction peaks corresponding to Ni and Ni3P were observed in the XRD patterns of the samples heated up to 600oC. At lower temperature of heating only peaks from Ni12P5 and Ni3P were identified. This observation does not correlate with the result presented in [3] where Ni and Ni3P were formed simultaneously during the crystallization process. The observations of the surface morphology of Ni-P amorphous films showed, that there are spherical particles with holes distributed mostly at the borders of particles. These holes, probably related to hydrogen, disappear during the heating. The rough surface morphology can be an important factor that influences the process of crystallization. The results presented in this report are relevant to processes taking place in Ni-P films obtained by a subject needs more experimental and theoretical work. crucial for controlling the nanocrystalline structures Further investigations in this area are in progress. Acknowledgement This work was supported by The Polish Scientific Research Committee KBN (project No PBZKBN-013/T08/05). References [1] C.A. Schuh, T.G. Nieh, T. Yamasaki: Scripta Materialia Vol.46 (2002), pp. 735-740 [2] C.C. Koch, J. Narayan: J. Mater. Res. Soc. Proc. B5.1.1 (2001), pp. 634 [3] Hexing Li, Haiying Chen, Shuzhong Wong, Jianshu Yang, Jing-Fa Deng: Applied Surface Science 125 (1998), pp. 115-119 [4] S. Linderoth, S. Morup: J. App. Phys. 69 (1991), p. 5256 [5] M. Trzaska, A. Wyszyska: Surface Engineering No 4 (2001), pp. 35-41 [6] M.S. Greval, S.A. Sastri, B.H. Alexander: Termochimica Acta Vol. 14 (1976), p. 25 [7] K.H. Hur, J.H. Jeong, D.N. Lee: J. Mat. Sci. Vol 25 (1990), pp. 2573-2584 [8] K. Ziewicz, Z. K dzierski, J. Morgiel, A. Ziewicz: Metallurgy and Foundry Engineering Vol. 27 (2001), No 2, p. 237 7 the description of the microstructures and chemical reduction method. However, this The early stages of crystallization, which are , especially, should attract more attention.

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